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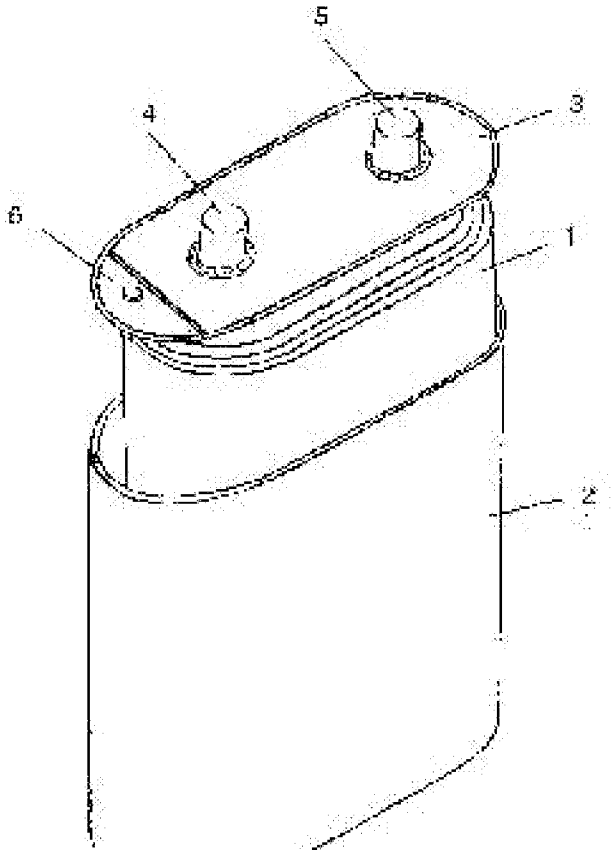
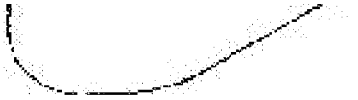
(54) NONAQUEOUS ELECTROLYTE SECONDARY BATTERY AND CAPACITY RESTORING METHOD THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a nonaqueous electrolyte secondary battery in which a part not opposed to a counter electrode for pasting a metal lithium to an electrode is not required, and lithium is evenly supplementary-charged over the entire electrode even if the electrode is long and large.

SOLUTION: The nonaqueous electrolyte secondary battery comprises a positive electrode, a negative electrode other than metal lithium, and a nonaqueous electrolyte. It is provided with a third electrode containing metal lithium which does not contact the electrolyte and is not connected to the positive electrode and the negative electrode.





JAPANESE [JP,2002-324585,A]

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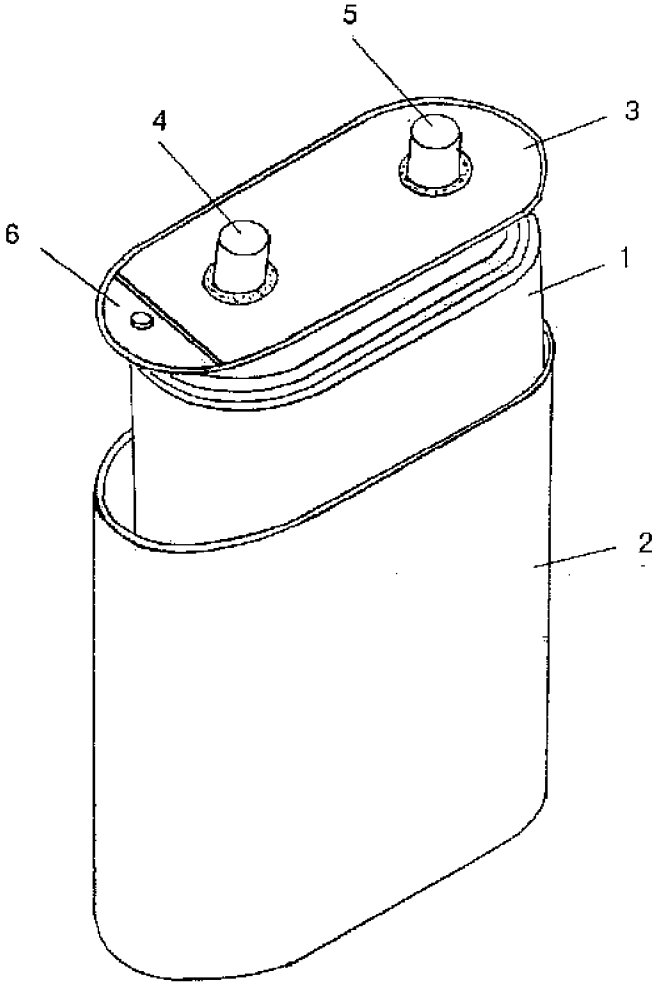
DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to a nonaqueous electrolyte secondary battery and the capacity method of recovery for the same.
[0002]
[Description of the Prior Art]The nonaqueous electrolyte secondary battery is widely used as a power supply of small size and a portable electronic device taking advantage of the feature of high energy density. Since the application to an electromobile will also be expected in the near future, development of a large-sized nonaqueous electrolyte secondary battery is recommended.
[0003]The nonaqueous electrolyte secondary battery is using the material which can emit [occlusion and] lithium for positive active material and negative electrode active material. The multiple oxide of transition metals, such as

Drawing selection Representative draw



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cobalt acid lithium, is used as positive active material, and, specifically, carbonaceous materials, such as lithium, a lithium alloy, and graphite, are used as negative electrode active material. By charge, occlusion of the lithium emitted from positive active material is carried out to negative electrode active material, by discharge, the lithium by which occlusion is carried out to negative electrode active material is emitted, and occlusion of the reaction of a nonaqueous electrolyte secondary battery is carried out to positive active material.

[0004]As an electrolysis solution of a nonaqueous electrolyte secondary battery, the nonaqueous electrolyte in which the mixed organic solvent containing various carbonic ester, such as ethylene carbonate and ethyl methyl carbonate, was made to dissolve lithium salt, such as $LiPF_6$ and $LiBF_4$, is used.

[0005]In such a nonaqueous electrolyte secondary battery, use carbon materials, such as black lead, for a negative electrode, and lithium to this carbon material occlusion and what is called a lithium ion battery made to emit, As compared with the cell which uses metal lithium, safety is high and, moreover, has the feature of being long lasting, with high energy density. Furthermore, in these days, what covered the surface of oxidized silicon (SiO), silicon (Si), or these particles with carbon is used as a negative pole material, and it has become clear to have the same feature as a carbon material.

[0006]

[Problem(s) to be Solved by the Invention]However, negative pole materials, such as what covered the surface of the carbon materials, oxidized silicon (SiO) and silicon (Si) of a nonaqueous electrolyte secondary battery, or these particles with carbon, react to the organic solvent and supporting electrolyte which constitute nonaqueous electrolyte. And an organic matter tunic is formed in the surface of these negative pole materials. It is known that the reaction which forms this organic matter tunic is a self-discharge reaction which consumes the lithium by which occlusion was carried out into the negative pole material.

[0007]After this self-discharge reaction assembles a nonaqueous electrolyte secondary battery and pours in organic electrolysis liquid, it advances continuously. When especially the nonaqueous electrolyte secondary battery was neglected by the charging state for a long period of time, the above-mentioned self-discharge reaction advanced, the lithium by which occlusion is carried out into the negative

pole material was consumed, the capacity of the cell fell, and it becomes impossible to have maintained the capacity balance of an anode and a negative electrode, and the charge-and-discharge reaction had a problem of ****** for trouble.

[0008]As one method for solving such a problem, metal lithium was directly stuck on the negative electrode, and what is called the "precharging method" was proposed as indicated, for example to JP,11-185809,A. [say / filling up a lithium source] However, in this precharging method, when metal lithium was stuck on the anode and the negative electrode which countered and charge and discharge were performed, the dendrite of lithium occurred in that portion at the time of charge, and there was a problem that an internal short circuit happened. The method of sticking metal lithium on the anode of a negative electrode and the portion (non-opposite portion) which has not countered directly had the dramatically long electrode like a large-sized nonaqueous electrolyte secondary battery, and when large, the whole electrode had a problem that lithium did not spread uniformly.

[0009]As indicated to the Patent Publication Heisei No. 509959 [11 to] gazette, or JP,10-270090,A, The 3rd electrode that equipped the inside of a cell with metal lithium or a lithium alloy beforehand is provided, it precharges by connecting this 3rd electrode and negative electrode, and the method of filling up the irreversible capacity of a negative electrode is also proposed.

[0010]Here, the irreversible capacity could not emit all the lithium by which occlusion was carried out into the negative electrode by initial charging when a carbon material was used for a negative electrode in a nonaqueous electrolyte secondary battery by discharge, but needed to put the thing of the lithium volume to which after discharge remains in a negative electrode, and this irreversible capacity needed to be decreased.

[0011]The method of using the 3rd electrode provided with conventional metal lithium and lithium alloy is for decreasing irreversible capacity, an equivalent for irreversible capacity is charged from the 3rd electrode at a negative electrode, and the 3rd electrode was used only before using a cell for the usual charge and discharge. Therefore, when the capacity lacks of the electrode of either the anode of a cell or a negative electrode arose after repeating a certain amount of charging and discharging cycle, capacity of the insufficiency was not able to be recovered.

[0012]When metal lithium is attached in the state where the

electrolysis solution was made to contact within a cell, like the above-mentioned example, When it offers beyond anticipation when capacity lowering is large, and the big space for metal lithium is required and long term storage is carried out,Even if an insulating film is formed in the interface of metal lithium and an electrolysis solution and it energizes to the 3rd electrode after that, current flows only through a few, There was a possibility that being eluted of lithium might become insufficient and there was a problem that an anode or a negative electrode, and the 3rd electrode contact inside a cell, and it would be in an overdischarge state in an anode, and they would be in an overcharging condition with a negative electrode further.

[0013]This invention solves such a problem, and does not need a non-opposite portion with the counter electrode for sticking metal lithium for an electrode, and like a large-sized nonaqueous electrolyte secondary battery, Also when an electrode is long and large, it aims at providing the nonaqueous electrolyte secondary battery which can perform the supplementary current of lithium uniformly to the whole electrode.

[0014]

[A means to solve a problem] In the nonaqueous electrolyte secondary battery provided with an anode, negative electrodes other than metal lithium, and nonaqueous electrolyte, including metal lithium, the invention of claim 1 did not contact an electrolysis solution, but was provided with the 3rd electrode that is not connected to an anode and a negative electrode.

[0015]According to the invention of claim 1, the nonaqueous electrolyte secondary battery which can carry out the supplementary current of the electrode to which service capacity fell is obtained.

[0016]Occlusion of the lithium is carried out to said one of electrodes by the invention of claim 2 being the capacity method of recovery of the above-mentioned nonaqueous electrolyte secondary battery, contacting the 3rd electrode to an electrolysis solution, and energizing to the electrode of either one of an anode or a negative electrode, and inter-electrode [3rd].

[0017]According to the invention of claim 2, the nonaqueous electrolyte secondary battery which does not need a non-opposite portion with the counter electrode for sticking metal lithium for an electrode, and can perform the supplementary current of lithium to the whole electrode uniformly also when an electrode is long and large is

obtained.
[0018]The invention of claim 3 was provided with two or more 3rd electrode in the above-mentioned nonaqueous electrolyte secondary battery. According to the invention of claim 3, the nonaqueous electrolyte secondary battery which can perform the supplementary current of lithium uniformly by the whole electrode is obtained.
[0019]In the above-mentioned nonaqueous electrolyte secondary battery, the invention of claim 4 is provided with a wound type power generation element, and the plate-like 3rd electrode, and is characterized by the flat surface of said the plate-like 3rd electrode being vertical to the winding medial axis of said wound type power generation element.
[0020]According to the invention of claim 4, the nonaqueous electrolyte secondary battery which can perform the supplementary current of lithium to the whole electrode uniformly easily is obtained.
[0021]
[Embodiment of the Invention]The large-sized nonaqueous electrolyte secondary battery which stored the wound type power generation element for the embodiment of this invention to the ellipse telescopic cell case is explained as an example. Generally as for the ability of capacity to apply truly the nonaqueous electrolyte secondary battery of 50 or more Ah to the cell whose capacity is smaller than this, as for this invention, either, a large-sized nonaqueous electrolyte secondary battery cannot be overemphasized here.
[0022]Drawing 1 - drawing 3 show the structure of the ellipse telescopic nonaqueous electrolyte secondary battery of this invention of using the cassette type 3rd electrode, The section structure of the cell in the state where attached the structure of the cassette type 3rd electrode to drawing 1, and the cassette type 3rd electrode was further attached to drawing 2 for the structure of the cell in the state where the cassette type 3rd electrode is not attached is shown in drawing 3.
[0023]In drawing 1 - drawing 3, 1 a power generation element and 2 a cell case and 3 A battery lid, 4 -- a positive pole terminal, a negative pole terminal and the door which 6 can open and close 5, and 7 -- the terminal of the 3rd electrode, and 8 -- as for metal lithium and 12, hermetic sealing and 10 are [an electrolysis solution and 14] external DC power a cassette case and 13 a collecting electrode plate and 11 the lid of the 3rd electrode, and 9.
[0024]The power generation element 1 overlaps an anode, a

separator, and a negative electrode on each other, and winds them around ellipse telescopic, ellipse telescopic metal casing is used as the cell case 4, and the battery lid 3 is insulated for the positive pole terminal 4 and the negative pole terminal 5 with hermetic sealing etc. As negative electrode active material, the material which can emit [occlusion and] other than metal lithium (for example, lithium, such as graphite) is used.

[0025]When the nonaqueous electrolyte secondary battery of this invention is carrying out charge and discharge in the usual state, the door 6 which can be opened and closed and which was provided in some battery lids 3 is closed. And the 3rd electrode is separated from a cell and the metal lithium 11 of the 3rd electrode is stored inside the cassette case 12. Therefore, lithium does not react to water etc.

[0026]When use this cell for a long time, the capacity of a negative electrode is consumed by self-discharge next, the capacity of a negative electrode decreases and the capacity balance between anode-negative electrodes collapses, As shown in drawing 3, take out the 3rd electrode from a cassette case and the door 6 which can open and close a cell is opened, If a supplementary current is energized and carried out in the direction which equip a cell with the 3rd electrode, and it is made for the metal lithium 11 to immerse into the electrolysis solution 13, and connects the terminal 7 and the negative pole terminal 5 of the 3rd electrode after that and in which a negative electrode is charged using the external power 14, The capacity of a negative electrode is recovered in the original state, and the capacity balance between anode-negative electrodes comes to be maintained. After the end of energization, the 3rd electrode is taken out from a cell, the door 6 which can open and close a cell is closed, the metal lithium 11 of the 3rd electrode returns to the state where it is stored inside the cassette case 12 and is not in contact in the electrolysis solution 13, and a cell is used for the usual charge and discharge.

[0027]If metal lithium 11 portion is stored to the cassette case 12 when the 3rd electrode is not used, the 3rd electrode can be used at any time, when required. It is necessary to perform wearing and extraction to extraction and storage, and the cell of the 3rd electrode from the cassette case of the 3rd electrode in atmosphere without moisture as it has stroked so that it may be dry room, in order to avoid adverse effects, such as moisture.

[0028]Drawing 4 and drawing 5 show the section structure of other examples of the nonaqueous electrolyte secondary

battery provided with the 3rd electrode of this invention, when, as for drawing 4, the cell is carrying out charge and discharge in the usual state, the capacity balance between anode-negative electrodes collapses and drawing 5 shows the case where the supplementary current of the negative electrode is being carried out using the 3rd electrode. In drawing 4 and drawing 5, the signs 1-14 show drawing 1 - the same thing as drawing 3, and 15 is a roller. As the 3rd electrode, what attached metal lithium was used for the band-like collecting electrode plate.

[0029]When the cell of this invention is carrying out charge and discharge in the usual state, as shown in drawing 4, the band-like collecting electrode plate 10 and the metal lithium 11 of the 3rd electrode are twisted around the roller 15, and the metal lithium 11 and the electrolysis solution 13 do not touch. Simultaneously, the terminal 7 of the 3rd electrode is not connected in the positive pole terminal 4 and the negative pole terminal 5. Therefore, in this state, the metal lithium 11 does not participate in a cell reaction.

[0030]When this cell is used for a long time, the capacity of the negative electrode was consumed by self-discharge next, the capacity of a negative electrode decreases and the capacity balance between anode-negative electrodes collapses, As shown in drawing 5, the band-like collecting electrode plate 10 and the metal lithium 11 are rewound from the roller 15, By making the metal lithium 11 immersed into the electrolysis solution 13, connecting the terminal 7 and the negative pole terminal 5 of the 3rd electrode after that, and energizing and carrying out a supplementary current in the direction in which a negative electrode is charged using the external power 14, the capacity of a negative electrode is recovered in the original state, and the capacity balance between anode-negative electrodes comes to be maintained. The 3rd electrode is rewound by the roller 15 after the end of energization, the metal lithium 11 returns to the state of drawing 4 which does not touch in the electrolysis solution 13, and a cell is used for the usual charge and discharge.

[0031]In this invention, especially as a structure of the 3rd electrode, it is not limited and can be considered as various shape, such as plate-like and rod form. Two or more number of the 3rd electrodes attached to a cell may be used with the size and capacity of not only one piece but a cell.

[0032]What is necessary is to connect an anode and the 3rd electrode and just to fill up the capacity of an anode, when the capacity of an anode decreases and the capacity balance

between anode-negative electrodes collapses contrary to a negative electrode although the above-mentioned example described the case where the capacity of a negative electrode decreased.

[0033]Next, the nonaqueous electrolyte secondary battery of this invention is provided with a wound type power generation element, and the winding medial axis of this wound type power generation element is parallel to the effective area of a square-shaped cell case, In the case of the structure inserted so that the flat part of a wound type power generation element and the effective area of a cell case might furthermore become vertical, as the 3rd electrode, it is considered as plate-like, and the relation between a wound type power generation element and the 3rd electrode has [making it the flat surface of the plate-like 3rd electrode be a perpendicular at the winding medial axis of a wound type power generation element] preferred things.

[0034]Drawing 6 is the thing in such a cell for which the relation between a wound type power generation element and a cell case was shown, and, as for a cell case and 16, 1 is [the winding medial axis of a wound type power generation element and 18] the flat parts of a wound type power generation element the effective area of a cell case, and 17 a wound type power generation element and 2 in drawing 6. In the square-shaped nonaqueous electrolyte secondary battery of this invention, the wound type power generation element 1 is inserted in the direction of the arrow shown in drawing 6 to the cell case 2. That is, the winding medial axis 17 of this wound type power generation element 1 is parallel to the effective area 16 of a square-shaped cell case, and it is inserted so that the flat part 18 of a wound type power generation element and the effective area 16 of a cell case may become vertical further. By considering it as such a structure, damage to the end face of the electrode at the time of inserting a wound type power generation element in a cell case is prevented, and a cell without an internal short circuit is obtained.

[0035]When the 3rd electrode, an anode, or a negative electrode is energized by considering a wound type power generation element, and the plate-like 3rd electrode as arrangement whose flat surface of the plate-like 3rd electrode is vertical to the winding medial axis of a wound type power generation element, a lithium ion is supplied to a power generation element from a direction parallel to the winding medial axis of a wound type power generation element. On the other hand, when it has arranged so that the

flat surface of the plate-like 3rd electrode may become a flat-surface part of a wound type power generation element, and parallel, Since it is not supplied to a power generation element whether a lithium ion is directions vertical to the flat-surface part of a wound type power generation element but it is supplied from a direction constantly parallel to the winding medial axis of a wound type power generation element, it must stop having to impress high tension from an external power.

[0036]Although the above-mentioned explanation explained only the case where negative electrode active material was a carbonaceous material, this invention is effective also when using the negative electrode active material which has the same irreversible capacity as carbonaceous materials, such as a tin oxidation thing, a silicon oxide, or silicon, besides this as negative electrode active material.

[0037]The power generation element of various shape, such as a wound type, a folded type, and a stack type, can be used for the shape of the power generation element of the nonaqueous electrolyte secondary battery of this invention. As shape of a cell, a square shape, cylindrical, ellipse telescopic, etc. can use the cell of all shape. Although it is usable irrespective of the capacity of a cell in this invention, comparatively generous capacity is effective in the space inside a cell especially in the large-sized nonaqueous electrolyte secondary battery of 50 or more Ah.

[0038]In the cell of this invention, as a solvent of nonaqueous electrolyte, Ethylene carbonate (EC), propylene carbonate (PC), Dimethyl carbonate (DMC), diethyl carbonate (DEC), Gamma-butyrolactone, sulfolane, dimethyl sulfoxide, acetonitrile, Polar solvents, such as dimethylformamide, dimethylacetamide, 1,2-dimethoxyethane, 1,2-diethoxyethane, a tetrahydrofuran, 2-methyltetrahydrofuran, dioxolane, and methyl acetate, or these mixtures may be used.

[0039]As a salt which nonaqueous electrolyte is made to contain furthermore, $LiPF_6$, $LiPF_3(C_2F_5)_3$, Lithium salt, such as $LiBF_4$, $LiAsF_6$, $LiClO_4$, LiSCN, LiI, $LiCF_3SO_3$, LiCl, LiBr, and $LiCF_3CO_2$, or these mixtures may be used.

[0040]As further a positive pole substance that can occlusion emit an alkaline metal, As an inorganic compound, the multiple oxide, the oxide which has a tunnel form hole, and the metal chalcogen ghost of the layer structure which are expressed with empirical formula Li_xMO_2 or $Li_yM_2O_4$ (however, M a transition metal,

0<=x<=1, 0<=y<=2) can be used.As the example, $LiCoO_2$, $LiNiO_2$, $LiMn_2O_4$, $LiMnO_2$, MnO_2, FeOOH, FeO_2, V_2O_5, V_6O_{13}, TiO_2, TiS_2, nickel oxyhydroxide, etc. are mentioned. As an organic compound, conductive organic polymer, such as poly aniline, etc. are mentioned, for example. Said various active materials may be mixed and used regardless of an inorganic compound and an organic compound.

[0041]In the cell of this invention, as a separator, polyolefin system separators with the conventional micropore, such as polyethylene and polypropylene, can be used, and it may be used combining these separators and solid polymer electrolytes. Only the solid polymer electrolyte having contained organic electrolysis liquid may be used without using a polyolefin system separator depending on the case. As a solid polymer electrolyte having contained this organic electrolysis liquid, a gel system solid polymer electrolyte, a porosity solid polymer electrolyte, etc. can be used.

[0042]

[Example]Hereafter, it explains using the suitable example of this invention.

[0043]Cobalt-acid-lithium ($LiCoO_2$) 80wt% as an active material, acetylene black 8wt% as an electric conduction auxiliary agent, and polyvinylidene fluoride (PVdF) 12wt% as a binder are mixed, N-methyl pyrrolidone (NMP) was added, it prepared to paste state, and this was applied to both sides of aluminium foil (150 mm in width, 1500 cm in length, and 20 micrometers in thickness), it dried at 100 **, NMP was evaporated, and the anode board with a thickness of 240 micrometers which equipped both sides with the active material layer was manufactured.

[0044]A negative electrode plate mixes graphite (black lead) 92wt% as an active material, and polyvinylidene fluoride 8wt% as a binder, N-methyl pyrrolidone (NMP) was added, it prepared to paste state, and this was made into both sides of copper foil (155 mm in width, 1550 cm in length, and 20 micrometers in thickness) with **, it dried at 100 **, NMP was evaporated, and the negative electrode plate with a thickness of 170 micrometers which equipped both sides with the active material layer was manufactured. As a separator, polyethylene fine porous membrane (160 mm in width and 40 micrometers in thickness) was used.

[0045]The lead terminal was welded to the end of the anode board and negative electrode plate which were obtained, respectively. The 100-micrometer-thick piece of nickel was

used for the negative electrode lead terminal at the positive electrode lead terminal using the 100-micrometer-thick aluminum piece. Then, both a positive electrode lead terminal and a negative electrode lead terminal wind, and make it become a part at first, and in this order, make it an anode board, a separator, a negative electrode plate, and separators overlap by turns, and they center on the winding core of the rectangular form of polyethylene, It wound around an ellipse whirl at the circumference, and was considered as the wound type power generation element with a size of 160x90x35 mm so that a long side might become a winding medial axis of a power generation element, and parallel.

[0046]Into the stainless case (190 mm in height, 95 mm in width, and 40 mm in thickness), this wound type power generation element was inserted, as shown in drawing 1, and the ellipse telescopic cell was assembled. The battery lid was equipped with the door portion which can be opened and closed and which inserts the 3rd electrode. And ethylene carbonate (EC) and diethyl carbonate (DEC) were mixed with the volume ratio 1:1, and it carried out vacuum pouring in of the electrolysis solution which added $LiPF_6$ of 1 mol/l.

Seal welding of a battery lid and the case was carried out after fixed time lapse, and the cell of nominal capacity 100Ah was manufactured.

[0047]As the 3rd electrode, the electrode whose sizes of the lithium portion which stuck 1-mm-thick lithium on the one side of a copper plate with a thickness [as a charge collector] of 20 micrometers same with having been shown in drawing 2 are 180 mm x 90 mm was used. This 3rd electrode is stored by the cassette case made from stainless steel when not using it.

[0048]First time charge of charging the same above-mentioned cell with the constant voltage of 4.1V continuously to 4.1V with the current of 1CA in **** for 20 cells and 25 ** for 2 hours was performed. Then, discharge is 50 cycle ********** about charge and discharge at the conditions of [continue to 2.0V with the current of 1CA, and charge continues to 4.1V with the current of 1CA, and] 2 hours at the constant voltage of 4.1V.

[0049]Then, ten cells of these 20 cells were divided into A group and each B group. About A group, the 3rd electrode was inserted for every end of 50 cycles, using the external power, only quantity of electricity which recovers the service capacity degraded minute from 1 cycle eye was energized in the direction in which a negative electrode is

charged by a negative electrode and inter-electrode [3rd], and the charge and discharge of the 50 following cycles were carried out to it. On the other hand, about B group, no 3rd electrode is used but the relation of the number of charging and discharging cycles and service capacity of the cell of A group and B group is shown in drawing 7 which continued charge and discharge as it was. In drawing 7, sign O is A group and sign - shows B group's cell. Service capacity of drawing 7 was made into the average value of ten cells each group's cell.

[0050]As shown in drawing 7, in A group's cell, the service capacity of 1 cycle eye is 50Ah, but the service capacity of 50 cycle eye is decreasing to 48Ah, and a service capacity degraded minute is 2Ah. Then, energization (equivalent to 2Ah of a service capacity degraded minute) was carried out in the direction in which a negative electrode is charged by 0.01CA (0.5A) for 4 hours, and it made a negative electrode and inter-electrode [3rd] recover negative-electrode service capacity to the same 50Ah as 1 cycle eye after the discharge end of 50 cycle eye. Similarly, by 0.01CA (0.5A), since the service capacity degraded minute of the 100 cycle eye was 2.5Ah, since the service capacity degraded minute of the 150 cycle eye was 3.5Ah about energization again, 0.01CA (0.5A) performed energization for 7 hours for 5 hours.

[0051]After energizing the service capacity of the cell of A group who is a cell of this invention to it having been alike, therefore service capacity having decreased [from which the number of charging and discharging cycles increases] using the 3rd electrode, by B group's cell, it was shown that service capacity is recovered, so that clearly from drawing 7.

[0052]

[Effect of the Invention]The supplementary current of a fallen part of long term storage or the capacity at the time of a cycle over a long period of time can be carried out by the 3rd electrode provided with metal lithium. [this invention] In this invention, the 3rd electrode does not contact the electrolysis solution of a cell, except when using it for a supplementary current, and further, since it is connected with neither an anode nor a negative electrode, it does not change during preservation. The capacity of this invention which uses a long and big electrode is [as opposed to / especially / the large-sized nonaqueous electrolyte secondary battery of 50 or more Ah] effective.

[Translation done.]